भारतीय मानक

Indian Standard

IS 9359 : 2023

फोरेट जी एंकेपसुलेटिड — विशिष्टि

(दूसरा पुनरीक्षण)

Phorate G Encapsulated — Specification

(Second Revision)

ICS 65.100.10

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February 2023 **Price Group 6**

Pesticides Sectional Committee, FAD 01

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

Phorate G are used as soil and systemic insecticide for the control of insects, mites, and certain nematodes in agriculture.

This standard was first published in 1980 and subsequently revised in 1995. In the first revision, the amendments issued to the standard were incorporated and a modified method for test for determination of phorate content included in the standard. Subsequently, in the year 2020 vide S.O. 1196 (E) dated 20 March 2020, phorate was banned by Government of India for domestic use but continued to manufacture for export. In this revision, the standard has been brought out in the latest style and format of the Indian Standards, and references to Indian Standards wherever applicable have been updated. It also incorporates one amendment issued to this standard.

In the preparation of this standard due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the rules framed thereunder and *Standards of Weights and Measures* (*Packaged Commodities*) *Rules*, 1977. However, this standard is subject to the restrictions imposed under these, wherever applicable.

The composition of the committee responsible for the formulation of this standard is listed in Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PHORATE G ENCAPSULATED — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for phorate G, encapsulated.

2 REFERENCE

The standards, given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards:

IS No.	*Title*
IS 460 (Part1) : 2020	Test sieves —– Specification: Part 1 Wire cloth test sieves (*fourth revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
IS 7976 : 2023	Phorate, technical —– Specification (*first revision*)
IS 8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 1 Solid pesticides (*second revision*)
IS 10627 : 1983	Methods of sampling of pesticides formulations

3 REQUIREMENTS

3.1 Constituents

The material shall consist of phorate, technical (*see* IS 7976) impregnated on suitable carrier(s) together with binder(s), stabilizer(s) and/or other formulants.

3.2 Physical

The material shall comply with the physical requirements specified in **3.2.1** to **3.2.5.**

3.2.1 *Description*

The material shall consist of dry, free flowing granules, free from visible extraneous matter and dust except for the amount specified (*see* **3.2.3**). It may contain added coloring matter.

3.2.2 *Sieving Requirement*

Not less than 97 percent by mass of the material shall pass through a test sieve having a mesh size of the upper declared limit and not more than 5 percent by mass of the material shall pass through a test sieve having mesh size of lower declared limit when determined by the method described in IS 6940.

3.2.2.1 The ratio of lower to upper sieve sixes declared under **3.2.2** shall not exceed 1 : 3.

3.2.3 *Dust*

When tested by the method prescribed in IS 6940, not more than 1 percent by mass of the material shall pass through 75 micron IS Sieve [*see* IS 460 (Part 1)]. The material retained on the sieve shall also comply with the requirements of **3.3.1**.

3.2.4 *Encapsulation*

3.2.4.1 When the material is subjected to the attrition test as prescribed in Annex A, the remnants shall not contain more than 2 percent by mass of declared nominal active ingredient.

3.2.4.2 When the material is subjected to the water run-off test as described in Annex B, the active ingredient in the water shall not exceed 5 percent by mass of the declared nominal active ingredient.

3.2.5 *Moisture Content*

When tested by the method prescribed in IS 6940, the moisture content of the material shall not exceed 4 percent by mass.

3.3 Chemical

The material shall comply with the chemical requirements specified in **3.3.1** and **3.3.2.**

3.3.1 *Phorate Content*

Phorate content shall be declared. When tested by the method prescribed in Annex C or Annex D. the observed phorate content percent (*m/m*) of any of the samples shall not differ from the declared nominal value by more than the tolerance, applied to the declared nominal value as given below (*see* **6.1** also).

Nominal Value, percent	*Tolerance, percent*	
Up to 9	+10 -5	of the nominal value
Above 9 and below 50	±5	
50 and above	+5 -3	

3.3.2 *Acidity*

When tested by the method prescribed in IS 6940, the acidity (as H_2SO_4), of the material shall be not more than 0.5 percent by mass.

4 PACKING

The material shall be packed as per requirements given in IS 8190 (Part 1).

5 MARKING

5.1 The containers shall be securely closed and shall be bear legibly and indelibly the following information in addition to any other information as required under the *Insecticides Act*, 1968 and Rules framed thereunder:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Date of expiry;
f) Net quantity;
g) Nominal phorate content, percent (*m/m*);
h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

6.1 When bulk manufactured material is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 within 90 days of its manufacture. The criteria for conformity shall be as given in IS 10627. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627. However, the criteria for conformity shall be the limits of tolerances given under **3.3.1** of this standard.

7 TESTS

7.1 Tests shall be carried out by the methods prescribed under **3** above.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 3.2.4.1)

METHOD FOR ATTRITION TEST

A-l Take 100 g of the material and sieve it for 2 hours in 75 micron IS sieve [*see* IS 460 (Part 1)] in a ro-tap machine.

A-2 Collect the remnants and determine its active ingredient as described Annex C.

ANNEX B

(*Clause* 3.2.4.2)

METHOD FOR WATER RUN-OFF TEST

B-l Take 10 g of the material in a 100 ml capacity burette plugged with cotton/glass wool at the bottom and add 50 ml water. Keep it for 15 minutes. Drain off the water after 15 minutes.

B-2 Determine active ingredient in the drained water so collected (*see* **B-l**) by the method described in Annex C.

ANNEX C

(*Clause* 3.3.1)

DETERMINATION OF PHORATE CONTENT

C-1 PRINCIPLE

C-1.1 Phorate is hydrolyzed in slightly aqueous acid in the presence of standard silver nitrate solution. The hydrolyzed product consume two moles of silver. The excess silver is titrated with standard ammonium thiocyanate solution. A correction for the amount of standard silver nitrate solution consumed by impurities present before hydrolysis is applied. Total silver consumed gives a measure of the active ingredient content.

C-2 REAGENTS

C-2.1 Acetone

C-2.2 Nitric Acid — Concentrated**.**

C-2.3 Nitrobenzene

C-2.4 Ferric Nitrate Solution — 10 percent.

C-2.5 Standard Silver Nitrate Solution — 0.1 N.

C-2.6 Standard ammonium Thiocyanate Solution — 0.1 N.

C-2.7 Standard Potassium Hydroxide Solution — 0.1 N, aqueous in 10 percent potassium nitrate.

C-2.8 Congo Red Test Paper

C-2.9 Benzene

C-3 PROCEDURE

C-3.1 Weigh 25 g of the sample and transfer the same to 250 ml iodine flask. Add 150 ml of acetone and shake well for 20 minutes. Filter the extract into a 250 ml volumetric flask. Add 50 ml of acetone in the iodine flask and shake for 15 minutes. Filter the extract in the same volumetric flask through the cotton plug. Repeat this treatment with 40 ml of acetone and transfer the extract into the same volumetric flask. Make up the volume with acetone and mix well.

C-3.1.1 Place 150 ml water, 5 drops of concentrated nitric acid and exactly 50 ml of standard silver nitrate solution into a 500 ml iodine flask. Immerse in a water-bath maintained at 50 °C until a constant temperature is attained. Pipette 50 ml of diluted sample (**C-3.1**) into iodine flask kept in water-bath; swirling the later during addition. Stopper and allow to remain in the 50 °C water-bath for 15 minutes and cool to room temperature. Add 5 ml nitrobenzene, stopper tightly and shake vigorously for about one minute. Add 1 ml of ferric nitrate solution, shake for a minute and titrate against standard ammonium thiocyanate solution. As the end point is approached, the solution will assume a light salmon-pink colour. Stopper and shake vigorously. The solution colour disappear. Add more of the titrant and again shake. Repeat this process until the colour is not completely discharged on shaking, but avoid adding excess of the titrant. When a little colour remains, remove the same by addition of a drop or two of standard silver nitrate solution. At this point, one additional drop of standard ammonium thiocyanate solution should reproduce the faint salmon-pink colour indicating end point for the titration.

C-3.2 Determination of Impurities

Take 50 ml of diluted solution (**C-3.1**) in a 500 ml separatory funnel containing 150 ml of benzene, extract the benzene solution thrice successively by shaking each time with 50 ml potassium hydroxide (*see* **C-2.7**) followed by 50 ml water, collecting the combined aqueous layers. Add nitric acid till acidic to congo red paper, which will turn blue. Add 5 drops of acid in excess, then add exactly 5 ml of standard silver nitrate, 5 ml of nitrobenzene and shake vigorously for about one minute. Add 1 ml of 10 percent ferric nitrate solution, shake for a minute and titrate with standard ammonium thiocyanate as described in **C-3.1.1**.

C-3.3 Calculation

C-3.3.1 Phorate content, percent by mass

$$= \left(\frac{65.10 \times (V_1 F_1) - (V_2 F_2)}{M_1} \right) - \left(\frac{65.10 \times (v_1 F_1) - (v_2 F_2)}{M_2} \right)$$

where

V_1 = volume, in ml, of standard silver nitrate solution consumed;
F_1 = normality of standard silver nitrate solution;
V_2 = volume, in ml, of standard ammonium thiocyanate solution consumed;
F_2 = normality of standard ammonium thiocyanate solution;
M_1 = mass, in g, of the material taken for the test (**C-3.1**);
v_1 = volume, in ml, of standard silver nitrate solution consumed for determination of impurities (**C 3.2**);
v_2 = volume, in ml, of standard ammonium thiocyanate solution consumed for determination of impurities (**C-3.2**); and
M_2 = mass, in g, of the material taken for determination of impurities (**C-3.2**).

ANNEX D

(*Clause* 3.3.1)

DETERMINATION OF PHORATE CONTENT

D-1 PRINCIPLE

The phorate, technical is determined by gas chromatography using internal standard technique.

D-2 APPARATUS

D-2.1 Gas Chromatograph (GLC) equipped with Flame Ionization Detector (FID) with facilities for on column injection and coupled to a printer-plotter-cum-integrator and a PC based data system is used for this determination. The suggested operative parameters are as follows, but can be changed, if necessary, provided standardization is done:

Column	Stainless steel, length = 1 m, ID = 2 mm, packed with 5 percent SE-30 on Chromosorb WHP (80 – 100 mesh)
Temperature	Column oven — 150 °C Detector — 250 °C Injector — 240 °C
Carrier Gas	Nitrogen — 30 ml/min Hydrogen — 30 ml/min Air — 300 ml/min
Retention time (Guide values)	Phorate — 5.56 min (Approx) Di-*n*-butyl phthalate — 3.68 min (Approx)

A typical chromatogram is given in Fig. 1 A

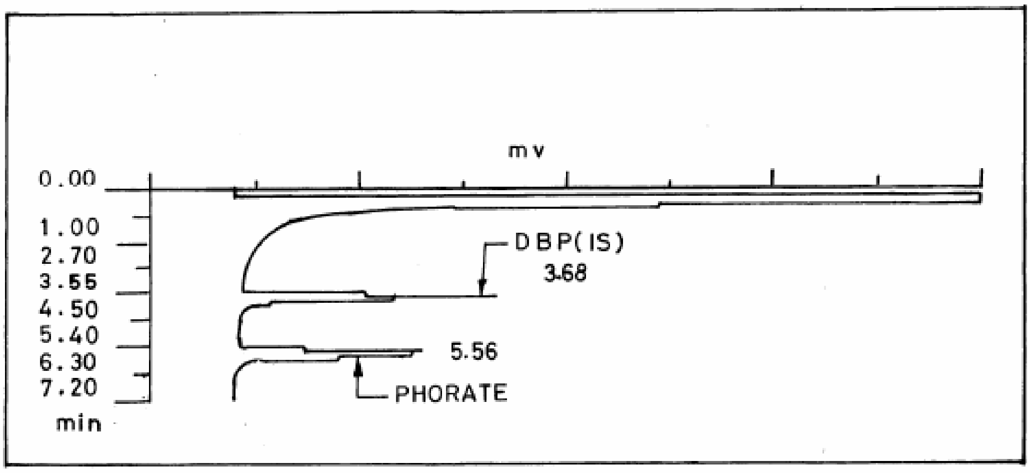

Fig. 1 A Typical Chromatogram

D-2.2 Injection Volume — 2 µl (microlitre).

D-2.3 Volumetric Flasks — 50 ml, 100 ml and 250 ml capacity.

D-2.4 Iodine Flask — 250 ml capacity.

D-2.5 Measuring Cylinder — 50 ml and 250 ml capacity.

D-2.6 Pipette — 5 ml and 10 ml capacity.

D-2.7 Microlitre Syringe — 10 µl capacity syringe with a needle of sufficient length to introduce the sample close to the top of the column packing.

D-3 REAGENTS

D-3.1 Acetone — AR grade.

D-3.2 Internal Standard — Di-n-butyl phthalate (DBP), AR grade or equivalent.

D-3.3 Phorate Reference Standard — of known purity.

D-4 PROCEDURE

D-4.1 Preparation of Internal Standard Solution

Weigh out accurately about 1.0 g of Di-*n*-butyl phthalate into a 100 ml volumetric flask. Dissolve in acetone and make up the volume up to the mark with acetone.

D-4.2 Preparation of Standard Solution

Weigh out accurately about 0.1 g of Phorate reference standard of known purity into a 50 ml volumetric flask. Add 5 ml of internal standard solution (**D-4.1**) and make up the volume up to 50 ml mark with acetone. Shake well to homogenize.

D-4.3 Preparation of Phorate, Technical Sample Solution

Weigh out accurately a quantity of technical sample so as to contain 0.1 g of phorate into a 50 ml volumetric flask and proceed as described in **D-4.2**.

D-4.4 Preparation of Phorate Granule Sample Solution

D-4.4.1 *Extraction of Phorate Granules*

Weigh accurately about 25 g of sample and transfer to a 250 ml iodine flask. Add 150 ml of acetone and shake well for 20 minutes. Filter the extract into a 250 ml volumetric flask. Add 50 ml of acetone in the iodine flask and shake for 15 minutes. Filter the extract into the same volumetric flask through the cotton plug. Repeat this procedure with 40 ml of acetone and transfer the extract into the same 250 ml volumetric flask. Make up the volume with acetone and mix well. Keep aside to get a clear solution. Then pipette out 10 ml of clear solution into 50 ml volumetric flask and proceed as described in **D-3.3**.

D-4.5 Estimation

D-4.5.1 Inject 1 µl of the reference standard solution into the gas chromatograph followed by the sample solution with the help of syringe and measure the peak areas of standard and sample solutions. Compute the percentage of phorate in the samples.

D-5 CALCULATION

For each Injection Response ratio, R =

$$\frac{\text{Phorate peak area}}{\text{Internal standard peak area}}$$

D-5.1 Phorate, Technical

Phorate content, percent by mass = $\frac{R_2}{R_1} \times \frac{W_1}{W_2} \times P$

where

R_1 = mean response ratio of standard solution;

R_2 = response ratio of sample solution;

W_1 = weight of reference standard in g;

W_2 = weight of sample in g; and

P = percentage purity of reference standard used.

D-5.2 Phorate Granules

Phorate content, percent by mass = $\frac{R_2}{R_1} \times \frac{W_1}{W_2} \times P \times D$

where

R_1 = mean response ratio of standard solution;

R_2 = response ratio of sample solution;

W_1 = weight of reference standard in g;

W_2 = weight of sample in g;

P = percentage purity of reference standard used; and

D = dilution factor for sample.

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

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Organization	*Representative(s)*
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